Researches on Multilayer Films with Polyethylene Core

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This paper has had as purpose rendering of some researches related to extrusion velocities while obtaining multilayer films with five layers of polymer. An installation comprising five extruders has been used for trials and conditions and optimal areas for every single extruder had been set. The results thus obtained made possible to settle the optimum parameters for the extrusion process so that to ensure a stable flow of the molten polymer, which resulted in a uniform multilayer, faultless structural film.

Keywords: polyethylene, extrusion, co-extrusion, multilayer film

Multilayer films have found large utilization in technics, thanks to their unique properties, resulted from the combination of multiple polymer layers within the same compound. The individual properties of polymers are determinant for their uses, some suitable for contact with food products, others posses barrier properties and others present compatibilizing properties for producing films free of the interlayer separation phenomena [1-2]. Multilayer films obtaining is done by extrusion. Those polymers should have identical viscosities, although there are known some cases when polymers with different viscosities have been successfully extruded [3-6].

In some cases, an interfacial instability can appear, causing a surface non-uniformity and layers mixture.

Flow instabilities that can appear within a multilayer melt on co-extrusion are of different types: a) non-uniform thicknesses starting from the extruder pulsation or from the melt thermal non-homogeneity [1, 4]; b) the more fluid polymer tend to migrate by shrouding the more viscous polymer; c) the layers intermixing becomes stronger with the increasing flow, i.e. the extrusion velocity when the fluid takes a ruffled aspect which is not visually observable from the beginning. The phenomenon becomes more accentuated with the increase of the velocity and it determines the formation of wrinkled surfaces; their multiplication gives the film a rough aspect known as the melt tear [4-8].

The same result of the melt tear is also obtained by decresing the temperature.

The above mentioned phenomenon diminishes down to its extinction by incresing the temperature, so the descrease of the extrusion velocity, i.e. the flow, or by choosing adequate polymers of the lower molecular mass and a narrow molecular mass distribution.

The experimental observations of this phenomenon lead to the conclusions that a critical interfacial shear stress (τ) exists, over which appears instability for a pair of polymers. Thus, for LDPE pairs of different molecular mass, τ_{crit}=30000 Pa, for LDPE polypropylene pairs, τ_{crit}=20000 Pa, and for HDPE polypropene, τ_{crit}=24000 Pa.

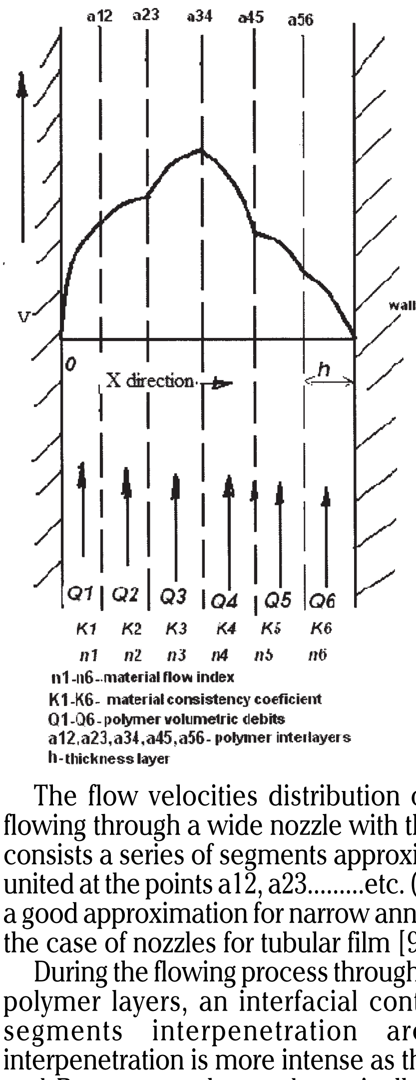

Fig.1. Schematic profile of the flow velocity of a non-Newtonian multilayer fluid through a pipe, where $\tau = K\gamma^n$, in which γ is the shear gradient

The flow velocities distribution of a multilayer fluid flowing through a wide nozzle with the opening of X = h consists a series of segments approximatively parabolical united at the points a12, a23.........etc. (fig.1) and represents a good approximation for narrow annular channels, as it is the case of nozzles for tubular film [9].

During the flowing process through nozzle of a two melt polymer layers, an interfacial contact and a polymer segments interpenetration are realized. This interpenetration is more intense as those two polymers A and B are more thermodynamically compatible, which corresponds to a structural resembling of [10-12].

The interface width increases by contact time, getting close to a limit value, which is higher as the polymers are more compatible [13-14].

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Cr. No.	Characteristic		LDPE	LLDPE	PA6	EVA	EVOH	OBS
1.	Densities, g/cm^3		0.930	0.920	1.12-1.13	0.930	1.16	
2	Glass transition temperature, °K/°C		-143.15/ -125	297/24	313.15/40	307.95/ 34.8	368.15/95	
3.	Melt Flow Index, MFI, (at 190°C,2,16kg) g/10min		0,3	2	-	2	2	
	Melt Flow Index, MFI, (at 210°C/2,16 kg), g/10min		-	-	-	-	4,1	
	Flow Index, ICT, (275°C/5kg)		-	-	$15cm^3/10$ min	-	-	
4.	Visco sity, Pa·s, at	190°C	$14,36x10^3$	$3x10^4$	-	$21,55x10^3$	$16,83x10^3$	
		195°C	$13,88x10^3$	$2,74x10^4$	-	-	$21,92x10^3$	
		210°C	$2,68x10^3$	$2,11x10^4$	-	$14,68x10^3$	$12,84x10^3$	
		220°C	-	-	-	$12,39x10^3$	-	
		225°C	$11,58x10^3$	-	$5,97x10^3$	$11,45x10^3$	-	
		227°C	$11,54x10^3$	-	-	-	-	
		230°C	$11,36x10^3$	-	$2,85x10^3$	$10,62x10^3$	-	
		235°C	$1,00x10^3$	$1,48x10^4$	$2,78x10^3$	-	$6,34x10^3$	
		275°C	-	-	$3,086x10^3$	-	-	
5.								

Table 1
MAIN CHACTERISTICS OF RAW MATERIALS

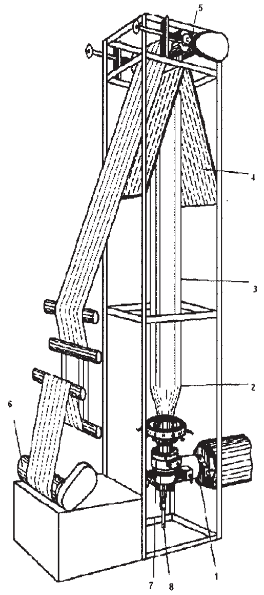

Fig.2. Film extrusion line for taking over the tube vertical wind [18]
1 - extruder, 2 - cooling plan, 3 - blown film, 4 - guiding panels; 5 - cylinder sealing film, 6 - film winding device;

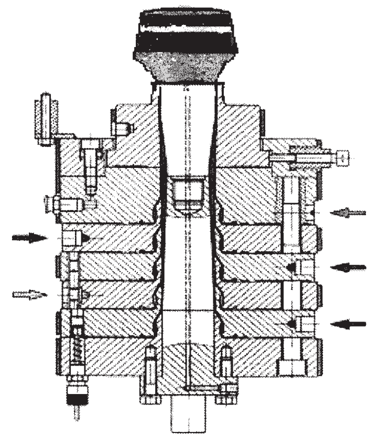

Fig.3. Co-extrusion head for 5 layers

When the co-extrusion of two different structural and thermodynamically non-miscible polymers is imposed, the single solution to avoid exfoliation is to introduce the third intermediary layer, compatible with both A and B polymers or with a copolymer block AB [11, 15-17].

An even better solution is the using of compounds or polar and/or grafted copolymers.

Their role is to optimize the interfacial adherence by combining the two polymers functionalities and to adjust the intermediary layer viscosity [18-24].

Low density polyethylene (LDPE), linear low density polyethylene (LLDPE), polyamide 6 have been used as polymers, while ethylene-vinyl acetate (EVA) and ethylene-vinil-alcohol (EVOH) copolymers as adhesives.

The purpose of the paper consists into setting the experimental conditions (temperatures, shear stresses, flows etc.) for an optimal extrusion of multilayer films without the instability phenomenon occurring.

Experimental part

Raw materials

The raw materials used have the characteristics presented in table 1.

The experimental determinations have been rendered by an instalation Omicron-type, equipped with five extruders with an extrusion- in-five-layers head presented as follows.

The co-extrusion head is similarly with the one prezented in figure 3.

The melt flow behaviour is described by Oswald de Waele law

$$\tau = K \dot{\gamma}^n \quad (1)$$

where τ *is the* shear stress, $\dot{\gamma}$ is the shear rate, K and n are constants for material (n-flow index; K-consistency index).

The Williams-Landel-Ferry model (WLF) [25-29] has been used for the dynamic viscosity calculus of polymers at different temperatures. This model has been expressed by the equation (2):

$$\eta(T) = \eta_0 \exp\left(\frac{-C_1 (T - T_r)}{C_2 + T - T_r} \right) \quad (2)$$

in which T- is the calculating temperature, °K, T_r- reference temperature,°K, C_1, C_2, η_0–constants, which when T_r equal T_g, have the values $C_1H \approx 17.44$; $C_2H \approx 51.6$; η_0, η – initial and final viscosity.

In order to determine the pressure and flowing velocity drops of every layer, it has been preestimated a material flow for every extruder and it has been taken into account the equation (3) of the pressure drop Δp at the nozzle wall with the relation [15]:

$$\Delta p = \left[\frac{Dv}{\pi R^3}\cdot\frac{3n+1}{n\cdot\dot{\gamma}^0}\right]^n\left(\frac{2\eta^0\cdot\dot{\gamma}^0\cdot L}{R}\right) \quad (3)$$

in which Δp – pressure drop, Pa; Dv – volumetric flow, m^3/s; R= 0.2 m – nozzle radius; η^0- dynamic viscosity, Pa.s; γ^o - velocity gradient at the reference state, which in this case is $1s^{-1}$; L=0.035m – nozzle length,

With the obtained value for Δp from the equation (3) introduced in the equation (4) of the pressure drop as a velocity function,

$$\Delta p = \xi\frac{w^2}{2\dot{\gamma}^0}\rho \quad (4)$$

the flowing velocity has been determined corresponding to every layer with the equation (5) [15]:

$$w = \sqrt{\frac{2\cdot\Delta p\cdot\dot{\gamma}^0}{\xi\cdot\rho}} \quad (5)$$

where:

$\zeta=1$, coefficient of local resistance for the case of fluids with sudden exstress;

ρ = density of material, kg/m^3.

The average velocity in the middle plane, which in fact is a correction of the flowing velocity, on every extruder, has been calculated with the relation (6)[15]:

$$w_m = \left(\frac{n+1}{2n+1}\right)w \quad (6)$$

where n has the following values [16-17]:
for LDPE 0.35; for EVA 0.40; for EVOH n=0.45, for LLDPE n=0.60 and for PA6 n=0.70.

The shear stress at the nozzle wall τ, in Pa, is calculated with the expression (7) [15]:

$$\tau = \frac{\Delta p}{2\left(\frac{L}{r}\right)} \quad (7)$$

Five types of multilayer films of the following compositions have been taken into consideration:

F1. 24.56%LDPE/11,59%EVA/27.7%PA6/11.59%EVA/24.56%LDPE corresponding to the thicknesses of 35 / 30/ 70 / 30 / 35μm, which by co-extrusion gives a total thickness of 200 μm

Table 2
WORKING PARAMETERS FOR OBTAINING THE MULTILAYER FILM: LDPE/EVA/PA6/EVA/LDPE

Working parameters	External Layer	Adhesive 1 Layer	Central Layer	Adhesive 2 Layer	Internal Layer	Extrusion on head
	LDPE Extruder 1	EVA Extruder 2	PA6 Extruder 3	EVA Extruder 4	LDPE Extruder 5	
Proposed film thicknesses, μm	45	30	70	30	45	220
Realized thicknesses, μm	43.2	29.5	68.7	29.7	44.1	215.2
Pressure in kgf/cm^2, in the entrance section (MPa)	170 (16.7)	190 (18.6)	320 (31.4)	117 (11.4)	250 (24.5)	319 (31)
Current Intensity to the extruder feeding, A	29	30	72	30	80	0,8
Screw velocities, rot/min	36	32	45	68	42	
Flow, kg/h (m^3/s)	53.4 ($1.595x10^{-5}$)	25.28 ($7.55x10^{-6}$)	59 ($1.46x10^{-5}$)	25.28 ($7.55x10^{-6}$)	53.4 ($1.595x10^{-5}$)	217.4 ($6.46x10^{-5}$)
Average melting temperatures,°C	210	210	225	225	230	227
Viscosity at the temperature at the nozzle entrance, Pas	$12.68\cdot10^3$	$14.68\cdot10^3$	$5.97\cdot10^3$	$11.45\cdot10^3$	$11.36\cdot10^3$	$18.43\cdot10^3$ (LDPE ext)
Density, Kg/m^3	930	930	1120	930	930	934.79 (Dm/Dv)
Exponent n, from the rel.(1) dimensionless	0.35	0.40	0.70	0.40	0.35	0.35 (LDPE)
Pressure drop in each feeder of the extruder, Pa	626.27	396.290	32.159	309.101	778.388	1485.186
Velocity at nozzle exit, m/s	1.16	1.29	0.239	0.815	1.1	1.78
Corrected velocity with the rel.(6), m/s	0.92	1.01	0.169≈ 0.17	0.63	0.87	1.41
Shear stress at nozzle wall, Pa (rel.7)	-	-	-	-	-	4246.24

Table 3
PHYSICO-CHEMICAL CHARACTERISTICS OF THE OBTAINED MULTILAYER FILM AT A TEMPERATURE OF 210-230°C AND PRESSURES OF 117-250 kgf/cm²

	Characteristics	UM	Testing method	Minimum values guidance for use compared to the majority of the materials	Achieved values
1.	Traction (pulling speed 100mm/min) MD- tensile strength - tension - elongation	 Kgf/cm² %	ISO 1184 :83	 min 61.18 -	 61.80 10%
	-to tear - resistance - elongation	 Kgf/cm² %		 min 142.7 200	 197.5 301
	TD-at stretching - tension - elongation	 Kgf/cm² %		 min 61.18 -	 61.80 12
	-to tear - resistance - elongation	 Kgf/cm² %		 102 250	 130 320
2.	Resistance to shock by traction/impact strength MD- for sheet -for material TD- for sheet - for material	 J/m kJ/m² J/m kJ/m²	ISO 6526, sample type 2 and 3	 - 1000 - 400	 209 2620 176 2225
3.	Resistance to impact Dartvis, multiaxially - impact strength at tear - impact energy at tear	 N J	ISO 7765-2	 min 50 -	 92 2.3
4.	Resistance at laceration MD-for sheet -for material	 N N/mm	ISO 6383-2	 min 2 -	 4.2 50
	TD-for sheet -for material	N N/mm		min 3 -	7.1 83
5.	Permeability to oxygen (23°C, 1 bar)	cm³/m²/24h	ISO 2556 :74	max 100	62
6.	Permeability to water wet air (23°C, saturation HR)	g/m²/24h		max 4	3.1
7.	Weight unit area	g/m²	ISO 4591 :92		204

F2. 18.75%LDPE/12.50%EVA/23.76%PE+EVOH(19.76% PE+4%EVOH)/18.74% EVA/26.25 % LDPE corresponding to the thicknesses of 30 / 20/ 50 / 25 / 35 μm, which by co-extrusion gives a total thickness of 160μm

Five types of multilayer films of the following compositions have been considered:

F3. 31,25% LLDPE / 6,25 % EVA / 25% PA6 / 6,25 % EVA / 31,25 % LLDPE corresponding to the thicknesses of 50 / 10/ 40 / 10 / 50 μm, which by co-extrusion gives the total thickness of 160 μm

Results and discussions

The viscosity, the pressure drops, the exit velocity for the nozzle, the corrected average velocity and the stress at the nozzle wall have been taken into consideration for the multiple layer film F_1, with 800 mm final width (table 2); the multilayer films F_2 have been similarly processed (tabele 4) as well as F_3 (table 6).

The analysis of the resulted characteristics, corroborated with the usage requirement shows that F_1 film can be used in storage and transport conditions for which higher physico-chemical characteristics of the film are necessary but without strict requirements for air permeability and moist air.

The use of LDPE+EVOH as a core layer compared to the use of only LDPE determines an increase of physical and mechanical properties, such as: tensile strength, impact strength by tensile, tear strength and low permeability to oxygen and humid air with water. These characteristics acquired by the addition of EVOH make them usable for packaging, storage and transport of perishable products which require limited moisture environments.

The use of PA 6 as a central layer compared to the use of LDPE+EVOH determined an increase of physical and mechanical properties, such as: tensile strength, impact strength by tensile, impact resistance, resistance to tear strength, and oxygen permeability to water and moist air are very close. These characteristics acquired by the addition of PA6 make them usable for packaging, storage and transport of perishable products that require tamper resistance and are subject to possible collisions.

In all analyzed cases, the pre-established volumetric and gravimetric flows have been initially used; the pressure gradients have been established by succesive experimental calculus and tests; the flowing velocity for an optimum extrusion have also been determined, no structural faults having been detected. The flowing velocities have been corrected with a correction coefficient, which takes into consideration the flowing index from power law, in order to obtain the flowing velocity of polymers in the average plan.

It is found that the determined velocities are influenced by the polymers nature and density, by the working pressure and the screw rotation.

Table 4
WORKING PARAMETERS FOR OBTAINING MULTILAYER FILMS CONTAINING ADHESIVE EVOH

Working parameters	External Layer	Adhesive 1 Layer	Central Layer	Adhesive 2 Layer	Internal Layer	Extrusion on head
	LDPE Extruder 1	EVA Extruder 2	LDPE+ adhesive EVOH Extruder 3	EVA Extruder 4	LDPE Extruder 5	
Proposed film thickness, µm	30	20	50	25	35	160
Realized thickness, µm	29.3	20.2	49.7	24.9	34.7	158.8
Pressure in kgf/cm2, in the feeder entrance section (MPa)	360 (35.3)	390 (38.25)	480 (47)	137 (13.43)	345 (33.83)	45
Melt temperatures, °C	225	225	195	220	195	235
Current Intensity to the extruder feeding, A	59	60	87	48	87	0,5
Screw rotations, rot/min	30	32	68	76	64	65
Volumetric flow, kg/h (m^3/s);	36 ($1.07x10^{-5}$)	24 ($7.17x10^{-6}$)	60 ($1.595x10^{-5}$)	30 ($8.96x10^{-6}$)	42 ($1.25x10^{-5}$)	192 ($5.61x10^{-5}$)
Viscosity at the temperature at the nozzle entrance, Pas	$11.58x10^3$	$11.45x10^3$	$13.88x10^3$	$12.39x10^3$	$13.88x10^3$	$11.00x10^3$
Density, kg/m^3	930	930	1040 (Dm/Dv)	930	930	945 (Dm/Dv)
Exponent n, from the rel.(1) dimensionless	0.35	0.40	0.37	0.40	0.35	0.35
Pressure drop in each feeder of the extruder, Pa	497.35	302.78	966.57	358.19	596.14	766.02
Velocity at the nozzle exit, m/s	1.03	0.81	1.36	0.88	1.13	1.27
Corrected velocity,m/s	0.82	0.63	0.89	0.68	0.89	0.84
Shear stress at nozzle wall, Pa, conf.rel.(7)	-	-	-	-	-	2188.63

Table 5
PHYSICO-CHEMICAL CHARACTERISTICS OF THE OBTAINED MULTILAYER FILM WITH EVOH AS ADHESIVE

	Characteristics	UM	Testing Method	Minimum Values as guidance for use, compared to the majority materials	Achieved Values
1.	Traction (pulling speed 100mm/min) MD-tensile strenght - tension - elongation	 Kgf/cm^2 %	ISO 1184 :83	 min 61,18 -	 89.6 7
	-to tear - resistence - elongation	Kgf/cm^2 %		min 142,7 200	207.3 245
	TD-in tension - tension - elongation	Kgf/cm^2 %		min 61,18 -	82.80 11
	-to tear - resistence - elongation	Kgf/cm^2 %		102 250	148 320
2.	Resistence at shock by Traction MD- for sheet -for material	J/m kJ/m^2	ISO 6526, sample type 2 and 3	- 1000	190 2300
	TD- for sheet - for material	J/m kJ/m^2		- 400	143 1825

3.	Resistence to impact Dartvis, multiaxially - impact strength to tear	N	ISO 7765-2	min 50	83
	- impact energy to tear	J			1.8
4.	Resistence at laceration MD -for sheet - for material	N N/mm	ISO 6383-2	min 2 -	4.4 49
	TD-for sheet -for material	N N/mm		- -	
5.	Permeability at oxygen (23°C, 1 bar)	$cm^3/m^2/24h$	ISO 2556 :74	max 100	33
6.	Permeability at water wet air (23°C, saturation HR)	$g/m^2/24h$		max 4	1.7
7.	Weight unit area	g/m^2	ISO 4591 :92		155

Table 6
WORKING PARAMETERS FOR OBTAINING THE FILM WITH THE STRUCTURE LLDPE/EVA/PA6/EVA/LLDPE

Working parameters	External Layer Extruder 1	Adhesive Layer 1 Extruder 2	Central Layer Extruder 3	Adhesive Layer 2 Extruder 4	Internal Layer Extruder 5	Extrusion on head
	LLDPE	EVA	PA6	EVA	LLDPE	
Proposed film thickness, μm	50	10	40	10	50	160
Realized thickness, μm	49.3	10.2	39.7	11.0	49.4	159.6
Pressures, in kgf/cm^2, at the entrance section; MPa	156 (15.3)	175 (17.1)	405 (40.2)	135 (13.2)	308 (30.2)	310
Average Melting temperatures, °C	210	210	225	220	190	235
Current intensity to the extruder feeding, A	28	29	79	45	70	0,5
Screw rotations,rot/min	23	23	57	89	62	65
Flow, kg/h	60	12	48	12	60	192
Volumetric flow, m^3/s	$1.8{\cdot}10^{-5}$	$3.6{\cdot}10^{-6}$	$1.12{\cdot}10^{-5}$	$3.6{\cdot}10^{-6}$	$1.8{\cdot}10^{-5}$	$5.48{\cdot}10^{-5}$
Viscosity at the temperature at the nozzle entrance, Pa·s	$2.11{\cdot}10^{4}$	$14.68{\cdot}10^{3}$	$5.97{\cdot}10^{3}$	$12.39{\cdot}10^{3}$	$3{\cdot}10^{4}$	$2.11{\cdot}10^{4}$
Density, kg/m^3	920	930	1120	930	920	972
Exponent n from the rel.(1) mensional	0.60	0.40	0.70	0.40	0.60	0.54
Pressure drop in each feeder, Pa	241.42	294.69	152.4	248.2	343.5	602.4
Velocity at the nozzle exit, m/s	0.72	0.79	0.52	0.73	0.86	1.11
Corrected velocity with the rel.(6), m/s	0.52	0.61	0.37	0.57	0.62	0.82
Shear stress at nozzle wall, Pa	-	-	-	-	-	1722.11

Table 7
PHYSICO-CHEMICAL CHARACTERISTICS OF THE OBTAINED MULTILAYER FILM WITH EVA AND PA6

	Characteristics	UM	Testing Method	Minimum Values as guidance for use, compared to the majority materials	Achieved Values
.	Traction (pulling speed 100mm/min) MD- tensile strength - tension - elongation	 Kgf/cm^2 %	ISO 1184 :83	 min 61.18 -	 92.6 7
	-to tear - strength - elongation	 Kgf/cm^2 %		 min 142.7 200	 217.3 255
	TD-in tension - tension - elongation	 Kgf/cm^2 %		 min 61.18 -	 84.60 11
	-to tear - resistence - elongation	 Kgf/cm^2 %		 102 250	 177 322

.	Resistence to shock by Traction		ISO 6526, sample type 2 şi 3		
	MD- for sheet	J/m		-	190
	-for material	kJ/m^2		1000	2335
	TD- for sheet	J/m		-	160
	- for material	kJ/m^2		400	1975
.	Resistence to impact Dartvis, multiaxially		ISO 7765-2		
	- impact strength to tear	N		min 50	87
	- impact energy to tear	J		-	1.4
.	Resistence at laceration MD-for sheet		ISO 6383-2		
	-for material	N		min 2	4.4
		N/mm		-	54
	TD-for sheet				
	-for material	N		min 3	6.9
		N/mm		-	87
.	Permeability at oxygen (23°C, 1 bar)	$cm^3/m^2/24h$	ISO 2556 :74	max 100	27
.	Permeability at water wer air (23°C, saturation HR)	$g/m^2/24h$		max 4	1.8
.	Weight unit area	g/m^2	ISO 4591 :92		123

Analyzing the experimental data, it is found that, for the high density polymers, as PA6, relatively low flowing velocities are needed (about. 0.17 m/s) of the film F1, at a feeding flow of 59 kg/h, in order to obtain a central film of thickness 70μm.

In the case of polymers used as adhesive interlayer (EVA), the flowing velocities are of 1.01m/s and 0.63m/s, at a flow of 25.28 kg/h, for a thickness of layer of 30μm.

The flowing velocities of PA6 (Film F_3) increase by the decrease of the flow and the layer thickness (0.37 m/s for a feeding flow of 48 kg/h and a layer thickness of 40μm), while the flowing velocities of the polar inter layers EVA decrease by the flow and the layer thickness decrease (0.61 and 0.57 m/s, for a flow of 12 kg/h and a layer thickness of 10μm).

This behaviour is explained by the important difference between the rheological parameters of those two polymers (table 2, 4.6).

It has been calculated the shear stress at the nozzle wall for every film apart. The more the shear stress at the nozzle wall is close to zero the polymer flowing through the nozzle is easier.

In all experiments performed, it has been noticed that the interfacial shear stress at the nozzle wall is below the critical value (for example, for the polyethylene sorts the critical value is of 0.0961MPa or 0.98 kgf/cm^2). Interfacial instability occurs at very close values or over the critical shear stress at the nozzle wall.

Conclusions

It is found that the corrected flowing velocities set in the average plane are influenced by the nature and density of polymers, by the working pressure and the screw rotation.

In all realized experiments, it was established that the interfacial shear stress at the nozzle wall is below the critical value, reason for which it was found to occur no interfacial instability phenomenon with the polymer breaking.

By experimental tests and calculus have been set the optimal flow of working of the extrusion installation in the case of the three types of multilayer film, without instability phenomenon occurring.

The physico-mechanical characteristics evidenced by tensile strength, impact strength by tensile, impact resistance, tear strength of films F2 with EVOH and of films F3 with PA6 are higher than those for F1 which have only PA6 in the inner layer. These characteristics acquired by the addition of PA6 make them usable for packaging, storage and transport of perishable products that require tamper resistance and are subject to possible collisions.

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